EXTRACTION OF WATER FROM ROCK/SOIL SAMPLES FOR ISOTOPIC ANALYSES

PROCEDURE ID: YMP-LBNL-TIP/TT-6.0	REV. 0, MOD. 0	EFFECTIVE: 09/30/98

1. PURPOSE

This Technical Implementing Procedure (TIP) describes a method to extract water for isotopic analyses from rock/soil samples, for the Yucca Mountain Site Characterization Project (YMP) at Lawrence Berkeley National Laboratory (LBNL).

2. SCOPE

This procedure shall be used by all LBNL personnel (or contractor personnel following LBNL procedures) involved in YMP activities whenever they are required to extract water from rock and/or soil samples for stable isotopic analyses. The procedures for analyzing the hydrogen and oxygen isotopic compositions of the water are contained in YMP-LBNL-TIP/TT 9.0, *Hydrogen Isotope Analyses of Waters*, and YMP-LBNL-TIP/TT 10.0, *Analysis of the Oxygen Isotopic Composition of Waters Using the Isoprep 18.* Prior to conducting work described in section 3.0 of this procedure, personnel extracting water from rock and/or soil samples require training in this procedure.

If this procedure cannot be implemented as written, YMP-LBNL personnel shall notify the responsible Principal Investigator (PI) or designee. If it is determined that a portion of the work cannot be accomplished as described in this TIP, or would produce undesirable results, that portion of the work shall be stopped and not resumed until this procedure is modified per YMP-LBNL-QIP-5.2, *Preparing Quality & Technical Implementing Procedures*.

If the responsible PI or designee determines that a modification or a revision to the TIP would cause an unreasonable delay in proceeding with the task, then an expedited change to the procedure, including documentation of deviation from the approved procedure, can be made according to YMP-LBNL-QIP-5.2. Such changes are subject to review, usually after the task has proceeded, and thus work performed under TIPs with expedited changes is done at risk of future invalidation.

Employees may use copies of this procedure printed from the controlled document electronic file; however, employees are responsible for assuring that the correct revision of this procedure is used. When this procedure becomes obsolete or superseded, it must be destroyed or marked “superseded” to ensure that this document is not used to perform work.

3. PROCEDURE

3.1 Principle

This procedure involves extracting water from soil and/or rock samples collected in the field by vaporizing the water in a vacuum and then distilling the water vapor into a trap chilled with liquid N_2 (LN). The water is then separated from other components that might also be extracted from the sample (primarily CO_2) by raising the temperature of the trap with a methanol slush (a mixture of LN and methanol adjusted to -80°C). This will cause impurities, such as CO_2, to be converted into a gas which can be pumped away, whereas the water will remain frozen in the trap. The water collected can then be stored for stable isotopic analyses (δD, $\delta^{18}O$). It is critical that almost all of the water be extracted from the sample (at least greater 95%) or the isotopic compositions of the water will be significantly fractionated (shifted) by the extraction process.

3.2 Materials/Equipment Required

- Balance (±0.1 g accuracy) calibrated in accordance with YMP-LBNL-QIP-12.0, *Control and Calibration of Measuring and Test Equipment*
- Glass vacuum line with flow-through traps (Attachment 1)
- Air-tight extraction vessel (Attachment 2)
- Glass beads
- Dewars
- Liquid N_2 (LN)
- Methanol
- Thermometer capable of measuring between 0 and -100°C.
- Water collection vial for storing water extracted from the sample
- Heat gun

3.3 Extraction of Water

3.3.1 After receiving the samples and prior to extracting the water, keep the sample stored in an airtight container (ideally the container used to collect the sample in the field) at 4°C. If the sample container is left open or broken in any way before extraction of the water, discard the sample.

3.3.2 Immediately prior to extraction of water from the sample, allow the container to warm to room temperature to avoid condensation of moisture onto the outside of the sampling container.

3.3.3 Weigh the sample and the container and record the weight in the laboratory notebook.

3.3.4 Fill both flow-through traps on the vacuum line (Attachment 1) approximately 30% full of glass beads (that have been thoroughly dried at >100°C). Seal the flow-through traps by placing an O-ring in the joint and clamping the joint. Prepare two methanol slushes by slowly adding LN to a dewar containing methanol until the desired temperature (-80 to -90°C in this case) is reached.

3.3.5 Attach a water collection vial to one of the ports on the right sample manifold of the vacuum line. The size of the collection vial should be large enough to hold the water in the sample (for Yucca Mountain samples, generally 5-10% of the sample size).

3.3.6 Evacuate the air in the flow-through traps and the sample collection vial by **slowly** opening the valves on the vacuum line leading to the vacuum pump (if the valves on the vacuum line are opened too quickly, the rush of gas to the vacuum pump will push the glass beads out of the flow-through traps and can break the glass line). Once the pressure in the traps has dropped to <1 millitorr (determined from the reading on the thermocouple gauges on the vacuum line), immerse both flow-through traps in dewars containing LN.

3.3.7 Place the sample (still in its original container) into an extraction vessel (Attachment 2) large enough to be sealed and attached to the vacuum line. Before placing the sample in the extraction vessel, be sure the outside of the sample container is thoroughly dry. Open the sample container and seal it inside of the extraction vessel, minimizing any contact between the sample and the ambient atmosphere. Connect the extraction vessel to one of the sample ports on the left manifold of the vacuum line.

3.3.8 Evacuate the extraction vessel by pumping the air through the flow-through traps and out through the right sample manifold. Once the pressure readings on the thermocouple gauges drop to <2 torr, heat the extraction vessel with a heat gun to facilitate transfer of the water out of the sample into the flow-through traps.

3.3.9 When the readings on **both** thermocouple gauges have dropped to <10 millitorr, close the valve on the sample port to which the extraction vessel is attached and remove the extraction vessel from the vacuum line. Weigh the extraction vessel with the sample still inside it and record the weight in the laboratory notebook.

3.3.10 Remove the dewars with LN from the flow-through traps and replace them with dewars containing the -80°C methanol slushes. Evacuate any gas that is evolved from the flow-through traps (predominantly CO_2) by pumping it out through the vacuum pump.

3.3.11 Once the reading on the thermocouple gauges have dropped to background levels, isolate the cold traps and the right sample manifold with the water collection vial by closing the valve leading from the left sample manifold to flow-through trap 1 and the valve above the right sample manifold that leads to the vacuum pump. Immerse the bottom half of the water collection vial in a dewar with LN and remove the methanol slushes from the flow-through traps, allowing the water in the traps to transfer into the water collection vial. When this process is complete (the reading on the thermocouple gauge 2 has dropped to background), close the valve above the water collection vial, remove the water collection vial from the vacuum line, seal it immediately and allow the sample to thaw. Record the yield of water in the laboratory notebook, label the water collection vial with the YMP Sample Management Facility (SMF) tracking number given the soil/rock sample when it was collected in the field and store it in the refrigerator for δD and $\delta^{18}O$ analyses.

3.3.12 **Caution -** If the amount of water in the sample is large (more than approximately 5 ml), the cold traps may become plugged with ice before all of the water has been extracted from the sample. When this happens, the reading on thermocouple gauge 2 (on the right sample manifold) will drop to background, whereas the reading on thermocouple gauge 1 (on the left sample manifold) will remain elevated. If this occurs, close the valve between the left sample manifold and flow-through trap 1 and the valve between the right sample manifold and the vacuum pump and transfer the water in the flow-through traps into the water collection vial (following steps 3.10-3.11 above). When this is done, cool the flow-through traps with LN again and continue distilling water out of the sample.

3.3.13 Records generated as a result of this TIP shall be entered into the appropriate YMP scientific notebook in accordance with YMP-LBNL-QIP-SIII.0, *Scientific Investigations*. Applicable elements of the laboratory notebooks are incorporated into the scientific notebook.

3.4 Acceptance Criteria

Proper completion of this procedure and submittal of records and associated data constitutes the acceptance criteria for this procedure.

4. RECORDS

4.1 Lifetime

Records generated as a result of this TIP are entries in scientific notebooks or attachments to such notebooks.

4.2 Non-Permanent

None

4.3 Controlled Documents

This Technical Implementing Procedure

4.4 Records Center Documents

Records associated with this procedure shall be submitted to Records Processing Center in accordance with AP-17.1Q, *Record Source Responsibility for Inclusionary Records*.

5. RESPONSIBILITIES

5.1 The **Project Manager** is responsible for final approval of the new, revised or modified TIP and for final approval of the rescission of the TIP.

5.2 The **EA Manager** is responsible for approving the new, revised or modified TIP, and for the rescission of the TIP.

5.3 The **OQA Representative** is responsible for reviewing and concurring with the TIP.

5.4 The **Principal Investigator (PI)** is responsible for assuring full compliance with this procedure and for providing training thereof. The

PI is also responsible for overseeing and coordinating the preparation, review, distribution, revision, and rescission of the TIP.

5.5 **Staff Members** are responsible for following this procedure and turning over related documentation to the Records Coordinator for submittal to the Records Processing Center in accordance with AP-17.1Q. Related data shall be turned over to the Technical Data Coordinator in accordance with YMP-LBNL-QIP-SIII.3, *Submitting Key Data to the Yucca Mountain Project Office*, who will be responsible for submitting key data to the Yucca Mountain Project Office for entry into the YMP Technical Data Base (TDB).

5.6 Document Control **Staff** are responsible for providing the controlled distribution of the TIP and modifications thereof.

6. ACRONYMS AND DEFINITIONS

6.1 Acronyms

EA Engineering Assurance

LBNL Lawrence Berkeley National Laboratory

LN Liquid nitrogen

OQA Office of Quality Assurance

PI Principal Investigator

SMF Sample Management Facility

TDB Technical Data Base

TIP Technical Implementing Procedure

YMP Yucca Mountain Site Characterization Project

6.2 Definitions

Staff Member: Any scientist, engineer, research or technical associate, technician, or student research assistant performing quality-affecting work for YMP-LBNL.

Technical Implementing Procedure: Each TIP describes YMP-LBNL technical tasks that (1) are repetitive, (2) are standardized, and (3) can return different results if deviation from the sequence of steps occur. TIPs are written when such tasks are sufficiently standardized to warrant

a formal procedure.

7. REFERENCES

AP-17.1Q, *Record Source Responsibility for Inclusionary Records*.

YMP-LBNL-QIP-5.2, *Preparing Quality & Technical Implementing Procedure*

YMP-LBNL-QIP-12.0, *Control and Calibration of Measuring and Test Equipment*

YMP-LBNL-QIP-SIII.0, *Scientific Investigations*

YMP-LBNL-QIP-SIII.3, *Submitting Key Data to the Yucca Mountain Project Office*

YMP-LBNL-TIP / TT 9.0, *Hydrogen Isotope Analyses of Waters*

YMP-LBNL-TIP / TT 10.0, *Analysis of the Oxygen Isotopic Composition of Waters Using the Isoprep 18*

8. ATTACHMENTS

Attachment 1 - Schematic diagram of the vacuum line used for extraction of water for isotopic analyses from rock / soil samples.

Attachment 2 - Schematic diagram of air-tight extraction vessel.

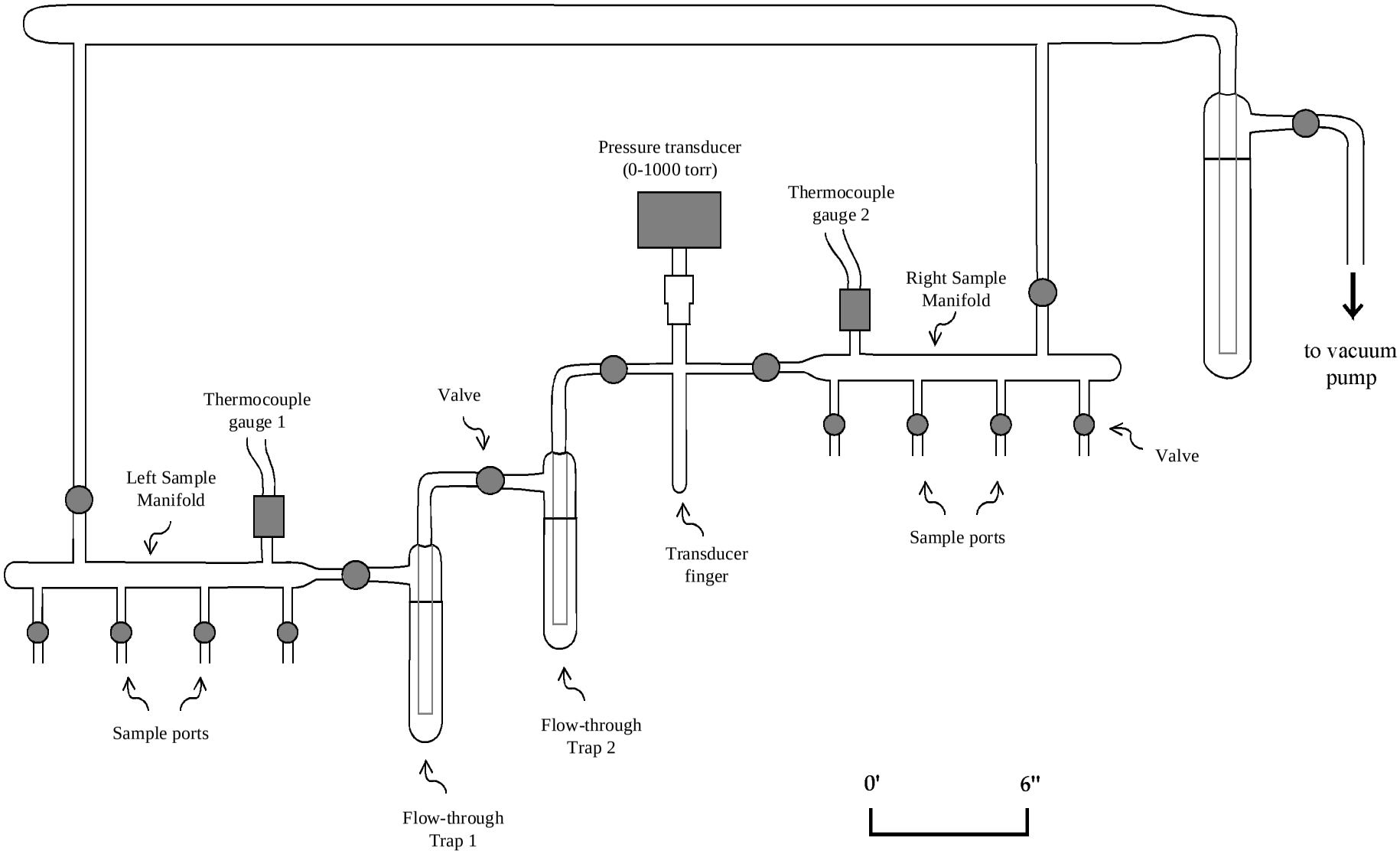
9. REVISION HISTORY

09 / 30 / 98 Revision 0, Modification 0

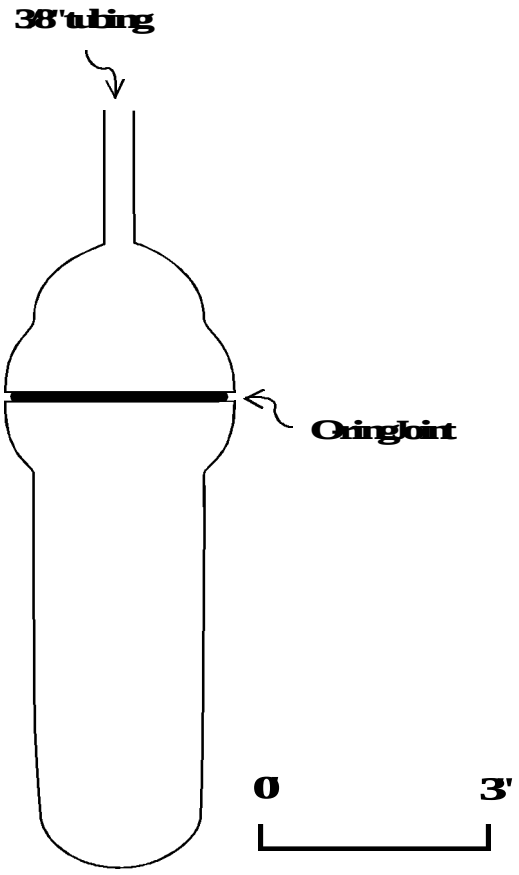
This is the initial issue of this TIP. This TIP is derivative a scientific notebook procedure / methodology entitled “Extraction of Water from Soil / Rock Samples for Isotopic Analyses” in Notebook YMP-LBNL-JSW-MC-1.

10. APPROVAL

Preparer: Mark Conrad	Date
Technical Reviewer: Nick Spycher	Date
Technical Reviewer: Eric Sonnethal	Date
EA Reviewer: Nancy Aden-Gleason	Date
OQA Concurrence: Stephen Harris	Date
Principal Investigator: Joseph Wang	Date
Project Manager: Gudmundur Bodvarsson	Date



Attachment 1 - Schematic diagram of the vacuum line used for extraction of water for isotopic analyses from rock/soil samples.



Attachment 2 - Schematic diagram of air-tight extraction vessel. Size shown is appropriate for a 125 ml (4 oz) wide-mouth Nalgene sample bottle.